

August 14, 1961

Mr. E. R. Helferich  
Watson Leavenworth Kelton & Taggart  
100 Park Avenue  
New York 17, New York

P. M. 374  
Addition of Nicotine to  
Tobacco Smoke

Dear Mr. Helferich:

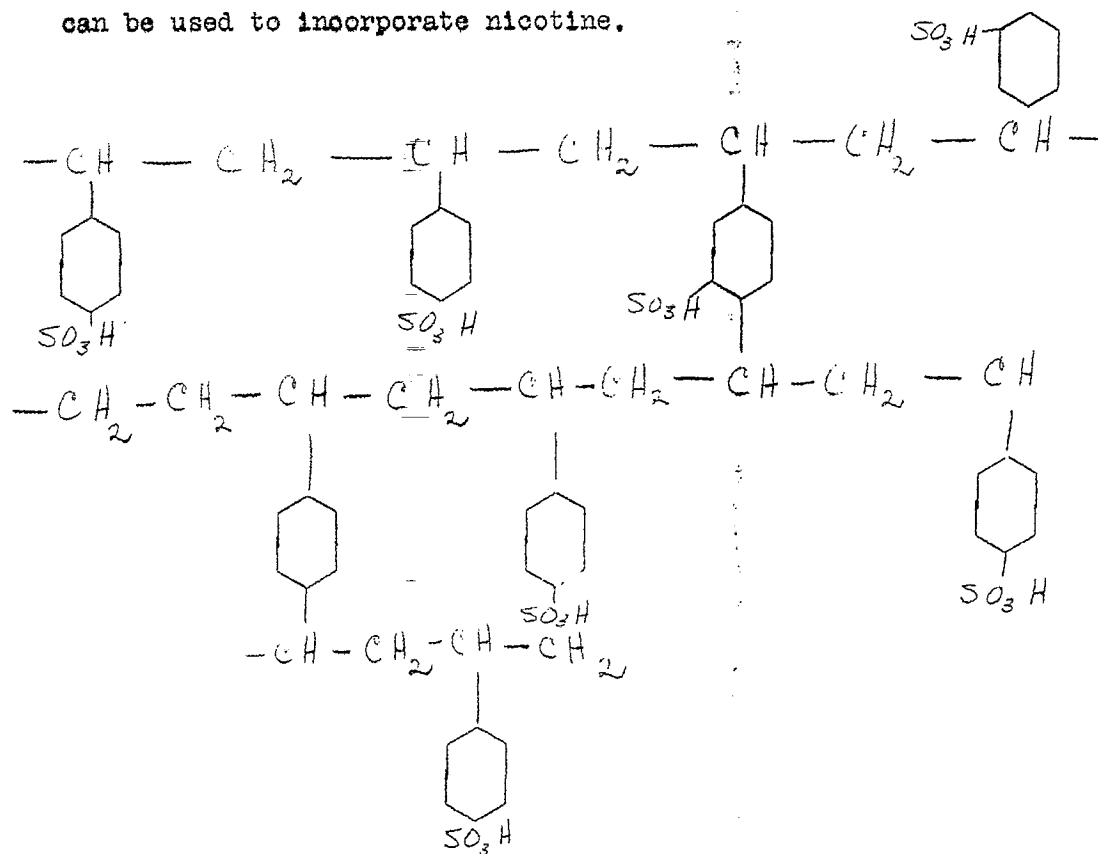
Information to Add to Disclosure of July 7, 1961

In a telephone conversation with you, you asked for generic descriptions of the resins used, chemical composition of the resins, range for addition of the nicotine to tobacco, additional examples of use of the resins, and information as to whether the nicotine was added as free nicotine or as nicotine salts, and whether nicotine derivatives could be used.

Additional work has been done using a strong cation ion exchange resin, a sulphonic acid type. These resins come under that classification: IR 112-H<sup>+</sup>, IR 120-H<sup>+</sup>, and Dowex 50 W-X8. Only IR 112 and Dowex 50 W-X8 have been used here. The structure of this type resin which is a sulphonated cross-linked polystyrene, cross-linked with various percentages of divinylbenzene, is given below: Any cation exchange resin having this basic structure

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can be used to incorporate nicotine.



Cigarettes were prepared using nicotine incorporated into Dowex 50W-X8 resin (Sulphonic acid type, a strong cation ion exchange resin of the structure given above). They were prepared to give a theoretical yield of 10 mg of resin per cigarette (22.8% of the treated resin was nicotine). The theoretical amount of nicotine would have been 2.3 mg/cigt. Chemical analysis of the tobacco filler showed that the actual nicotine content was 2.4 mg/cigt or 10.5 mg of the resin. The amount of nicotine present in the tobacco filler prior to the addition of the resin was 18.5 mg/cigt or 1.67%.

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Cigarettes were prepared using nicotine incorporated into IR 112 (sulphonic acid type, a strong cation ion exchange resin of the structure given below).

The cigarettes were prepared to give a theoretical yield of 20 mg of resin per cigarette (35.3% of the resin was nicotine). The theoretical amount of nicotine would have been 7.1 mg/cigt. Chemical analysis of the tobacco filler has shown that the actual nicotine content was 6.9 mg/cigt or 19.6 mg of the resin. The amount of nicotine present in the tobacco filler prior to the addition of the resin was 18.5 mg/cigt or 1.67%.

A structure for Amberlite CG-50 Type 1 - IR-50 (weakly acidic cation exchange resin, carboxylic type, hydrogen form) and the equation for the incorporation of nicotine were given on page 3 of the July 7 disclosure. The same equation represents the preparation of the nicotine-resin when any of the other types of resins are used.

Example 1 of that disclosure gave experimental details of its preparation and use. A general structure for a weak cation exchange resin having a carboxylic acid type functional group is given below. These resins are in  $H^+$  form and are prepared by co-polymerizing methacrylic acid. Amberlite CG 50, Type 1 (IR-50) used in Example 1 of July 7 disclosure is of this form. It is a polymer bead resin of the acrylic type. (See p. 122 - ION EXCHANGERS IN ORGANIC AND BIO-CHEMISTRY - Calmon, Kressman - INTERSCIENCE 1957, N. Y. N. Y.) They contain 4 - 6% divinylbenzene as a cross-linking agent.

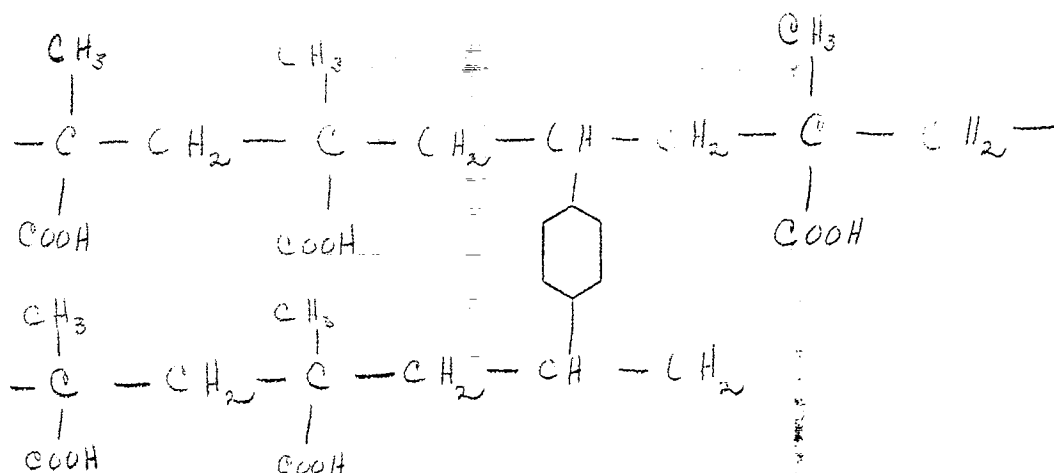
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Amberlite CG, Type 1

Weak Cation

Carboxylic Acid Type

Prepared by copolymerizing methacrylic acid with cross-linking agent.



Here is additional information on the theoretical and actual values of the nicotine content of the resin described in Example 1 of the July 7 disclosure.

Cigarettes were prepared to give a theoretical yield of 50 mg of resin per cigarette (27.3% of the resin was nicotine). The theoretical amount of nicotine would have been 13.6 mg/cigt. Chemical analyses of the tobacco filler showed that the actual nicotine content was 13.0 mg/cigt or 47.6 mg. of the resin. The amount of nicotine present in the tobacco filler prior to the addition of the resin was 14 mg/cigt or 1.27%.

The information contained in the 3 examples above has been consolidated into the following table. Ranges of addition of resin to tobacco filler and the resulting increase in the nicotine content of the smoke due to the incorporation of the nicotine-resin can be seen from the table. In this table, the nicotine content of the smoke

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has been increased in a range of 0.1 to 0.4. Claims should be made to cover a range of nicotine addition to smoke from 0.1 to 2.0 mg/cigt. The adjustment is made in the amount of resin added to the tobacco filler.

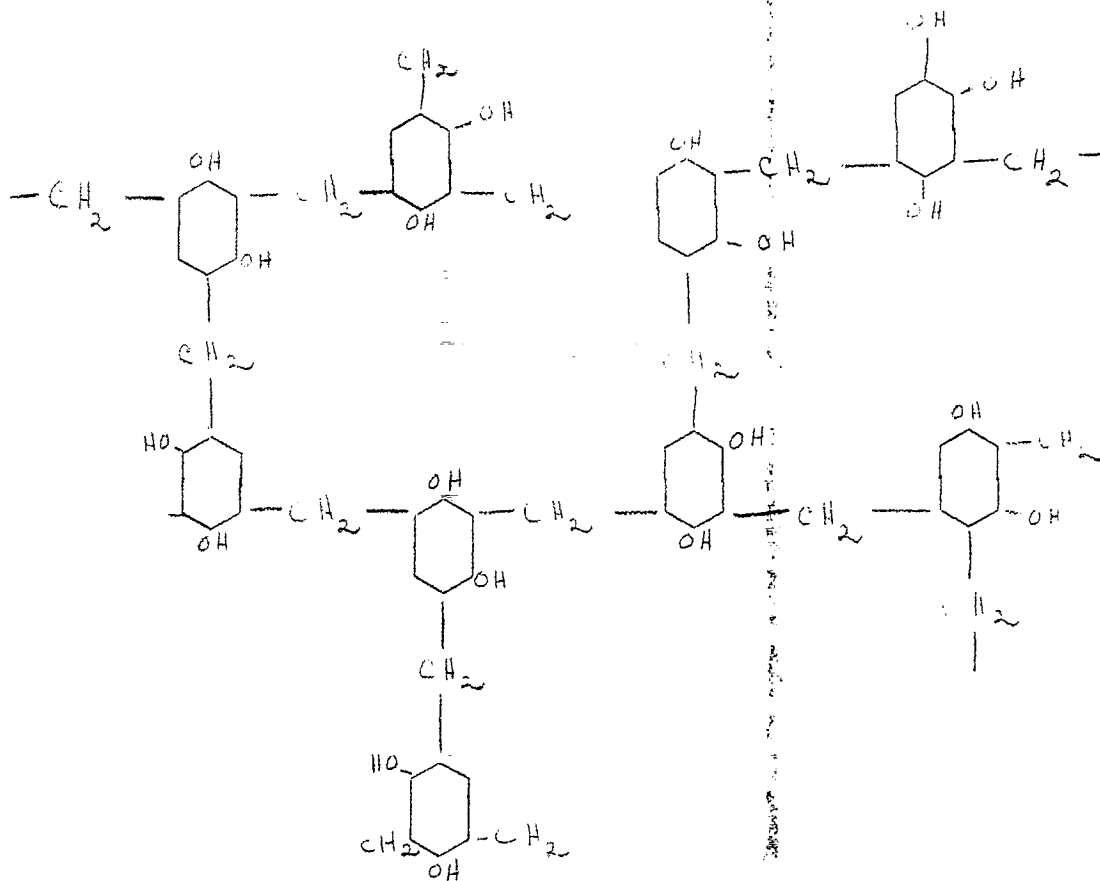
Cigt.Type	Nicotine in cigt. filler prior to the addition of resin	Theoretical amount of resin added	Actual amt. of resin added	Total nicotine content of cigt. filler	Nicotine in filler from resin	Total amt. of nicotine in smoke	Nicotine increase in the smoke due to the resin
Control #1	14.0	0	0	14.0	0	1.2	0
Control #1 plus Amberlite IR 50 resin	14.0	50	47.6	27.0	13.0	1.6	0.4
Control #2	18.5	0	0	18.5	0	1.2	0
Control #2 plus Dowex 50W resin	18.5	10	10.5	20.9	2.4	1.3	0.1
Control #2 plus Amberlite IR 112 resin	18.5	20	19.6	25.4	6.9	1.4	0.2
Control #3							

All values in mg/cigt.

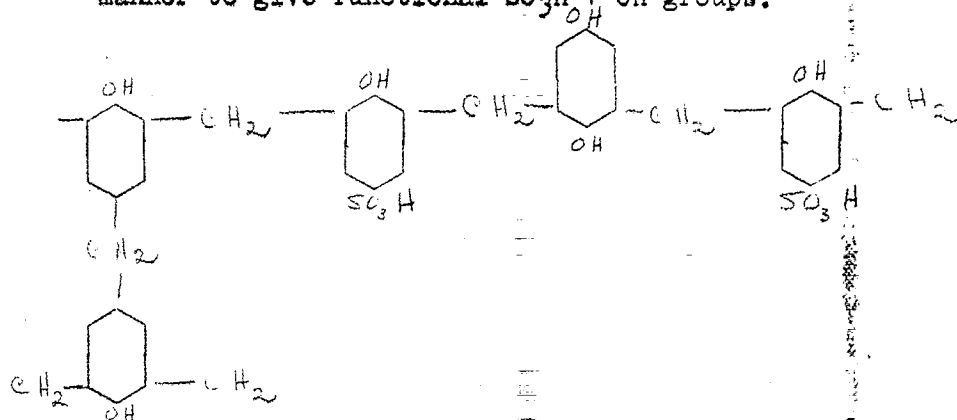
The structures given above and the information concerning the incorporation of nicotine are equally applicable to all available resins. Coverage should be obtained as broadly as possible on these resins. Structures of some additional types are given below. Weak cation resins containing

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one type of ion functional group - an OH group - are prepared by reacting polyhydric phenols with formaldehyde.

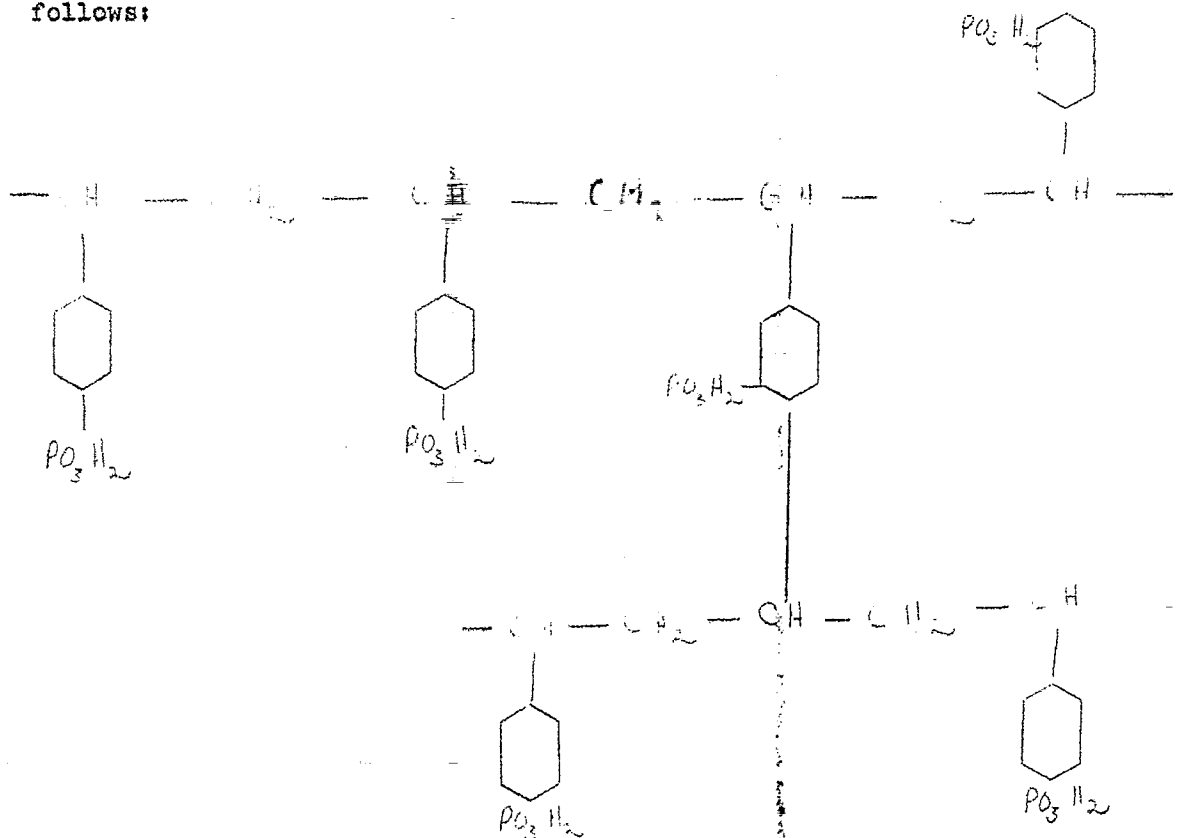


Sulphonic group can be introduced into a phenolic resin in this manner to give functional SO<sub>3</sub>H + OH groups.



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Another type of resin on which we will have experimental data of the type given above for the other resins is an intermediate acid, polystyrene type, containing a phosphonic acid difunctional group on a styrene - divinylbenzene lattice. The structure is as follows:



In the equation given in the disclosure of July 7, it is shown that free nicotine is used in the incorporation with the resin. This results in the formation of a nicotine salt. Nicotine derivatives could be used also but we have no specific data concerning their use. Upon pyrolysis of nicotine derivatives, the end product may not produce nicotine in smoke. It, therefore, would be better to ignore nicotine

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derivatives other than the nicotine salts formed as given in the examples above and in the former disclosure.

Sincerely yours,

*Thelma C. Heatwole*

(Mrs.) Thelma C. Heatwole  
Patent Liaison Officer

TCH:je

cc: Dr. A. Bayley  
Mr. Frank Resnik

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